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ILLINOIS UNIV AT URBANA-CHAMPAIGN DEPT OF CHEMISTRY
OZONOLYSIS OF DIPHENYLVINYLENE CARBONATE. THE SYNTHESIS AND THE--ETC(U)
DEC 79 G B SCHUSTER , J R HURST
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1. REPORT NUMBER N0014-76-C-0745-22	2. GOVT ACCESSION NO. 012	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) Ozonolysis of Diphenylvinylene Carbonate. The Synthesis and Thermolysis of Dibenzoylmonoperoxycarbonate.	5. TYPE OF REPORT & PERIOD COVERED Technical report	6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Gary B. Schuster and John R. Hurst	8. CONTRACT OR GRANT NUMBER(s) N00014-76-C-0745	9. PERFORMING ORGANIZATION NAME AND ADDRESS Department of Chemistry University of Illinois Urbana, IL 61801
10. CONTROLLING OFFICE NAME AND ADDRESS Chemistry Program, Materials Science Division, Office of Naval Research, 800 N. Quincy Street Arlington, VA 22217	11. REPORT DATE December 1979	12. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS NR 051-616
13. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)	14. NUMBER OF PAGES 15	15. SECURITY CLASS. (of this report) Unclassified
16. DISTRIBUTION STATEMENT (of this Report) This document has been approved for public release and sale; its distribution is unlimited LEVEL 1 12/15		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report) DDC RECEIVED DEC 14 1979 E		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Chemiluminescence Electron Transfer Kinetics Excited State Reactions 79 12 13 023		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Dibenzoylmonoperoxycarbonate (2) was prepared by the reaction of diphenylvinylene carbonate (1) with ozone. The thermolysis of 2 in benzene gives products typical of the formation of benzoyloxy radicals. The reaction rate was investigated at different temperatures and the activation parameters were determined ($\Delta H^\ddagger = 27.8 \pm 1.2$ kcal/mol, $\Delta S^\ddagger = 6.1 \pm 3.5$ eu). The thermolysis of 2 in the presence of the stable free radical galvinoxyl was investigated also. These experiments revealed that the fraction of radical escape from the solvent cage is $60 \pm 5\%$.		

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Contract NO014-76-C-0745

Task No. NR-051-616

TECHNICAL REPORT NO. NO014-76-C-0745-22

Ozonolysis of Diphenylvinylene Carbonate.

The Synthesis and Thermolysis of

Dibenzoylmonoperoxy carbonate

by

Gary B. Schuster and John R. Hurst

Prepared for Publication

in

Journal of the American Chemical Society

School of Chemical Sciences

University of Illinois

Urbana, Illinois 61801

December 6, 1979

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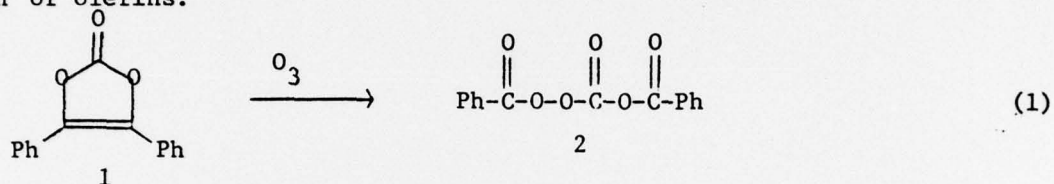
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Abstract:

(I)
Dibenzoylmonoperoxycarbonate (2) was prepared by the reaction of diphenyl-
vinylene carbonate (1) with ozone. The thermolysis of $\frac{I}{2}$ in benzene gives products
typical of the formation of benzoyloxy radicals. The reaction rate was investiga-
ted at different temperatures and the activation parameters were determined ($\Delta H^\ddagger =$
 27.8 ± 1.2 kcal/mol, $\Delta S^\ddagger = 6.1 \pm 3.5$ eu). The thermolysis of $\frac{I}{2}$ in the presence of
the stable free radical galvinoxyl was investigated also. These experiments
revealed that the fraction of radical escape from the solvent cage is $60 \pm 5\%$.

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Ozonolysis of sterically hindered olefins in some instances gives epoxides as well as ozonides and their derived products.² In contrast, our attempt to epoxidize the hindered olefin diphenylvinylene carbonate (1) with ozone gave the unusual rearranged peroxide dibenzoylmonoperoxycarbonate (2). Peroxycarbonate 2, to the best of our knowledge, is the first reported example of a diacylmonoperoxy-carbonate. Its formation is simply understood in terms of intramolecular trapping of the intermediate carbonyl oxide proposed by Criegee to be formed in the normal ozonation of olefins.³



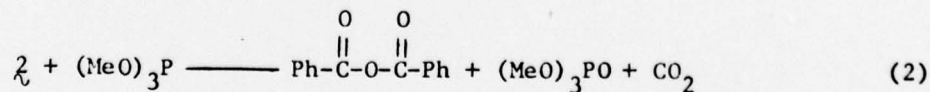
Peroxide 2 is related to benzoyl peroxide in that it can be prepared conceptually by inserting CO_2 between the carbonyl and phenyl groups of that well-known peroxide.⁴ The products of the thermolysis of 2 in benzene are, in fact, similar to those obtained from the thermolysis of benzoyl peroxide.^{5,6,7} However, the rate of reaction of 2 is significantly greater than that of benzoyl peroxide. The enthalpy and entropy of activation for the unimolecular decomposition of 2 suggest, moreover, that initial simultaneous two bond cleavage occurs in this system.

Kiefer and Traylor⁸ have concluded that formation of small molecules between the tert-butoxy radicals that result from the thermolysis of di-tert-butyl peroxide, di-tert-butyl hyponitrite, or di-tert-butyl peroxyoxalate decreases the ratio of in cage to cage escaped products. Comparison of benzoyl peroxide and 2 expand this study since a molecule of CO_2 is formed between the benzoyloxy radicals from 2.

RESULTS AND DISCUSSION

Synthesis. The addition of excess ozone to a solution of diphenylvinylene carbonate in CH_2Cl_2 gives, upon evaporation of the solvent, a peroxidic, colorless solid. In CCl_4 solution this product exhibits strong ir absorptions at 1853 cm^{-1} and 1787 cm^{-1} . Since no other diacyl monoperoxycarbonates have been reported, it is not possible to compare these absorptions with exact structural analogs. However, acyl carbonates have absorptions around 1800 cm^{-1} and 1740 cm^{-1} ⁹, and the modification of this structure to form 2 is expected to shift the carbonyl absorptions toward higher frequency. Thus, these two ir bands are consistent with structure 2. The ^1H NMR of the ozonolysis product in CDCl_3 reveals that all of the hydrogens are bound to aromatic rings. Importantly, the ^1H NMR shows two sets of doublets (4 protons) at 8.02-8.12 ppm and a multiplet (6 protons) at 7.35-7.70 ppm. The presence of the downfield doublets is indicative of benzoyl groups, and the fact that there are two doublets confirms that the benzoyl groups are nonequivalent. The ^{13}C NMR of the ozonolysis product in CDCl_3 shows 11 lines, three of which are in the carbonyl region (162, 158.9, and 146.9 ppm). The remaining lines are in the aromatic region (124.6 ppm to 135.2 ppm). The lines at 162.0 ppm and 158.9 ppm indicate nonequivalent benzoyloxy groups, and the lines at 146.9 ppm is consistent with a carbonate carbonyl group

Final conformation of the assigned structure for the ozonolysis product comes from its reaction with trimethyl phosphite. This reduction gives benzoic anhydride in 69% isolated yield.¹⁰



Thermolysis of 2. The thermolysis of monoperoxycarbonate 2 in benzene gives benzoic acid, biphenyl, phenyl benzoate, and CO_2 . In addition, there is a neutral, non-volatile residue that we assign as tetrahydroquaterphenyl.⁵ The product yields are detailed in Table 1. Basically, we can account, within experimental error, for all of the fragments of 2 among the products we detect.

The thermal decomposition of a 2.23×10^{-2} M solution of Z in benzene exhibits first order kinetics over the temperature range we examined (60.0-81.1°C). This observation, and the experiments carried out in the presence of the stable free radical galvinoxyl described below, rule out radical induced homolysis reactions under these conditions. Examination of the temperature dependence of the rate for the unimolecular thermolysis of Z , Table 2, reveals that $\Delta H^\ddagger = 27.8 \pm 1.2$ kcal/mol, and $\Delta S^\ddagger = 6.1 \pm 3.5$ eu. The entropy of activation, in particular, is consistent with an uncatalyzed unimolecular reaction, as is the relatively low value for the enthalpy of activation.¹¹

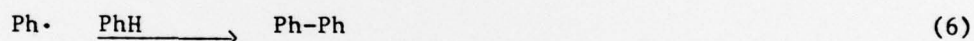
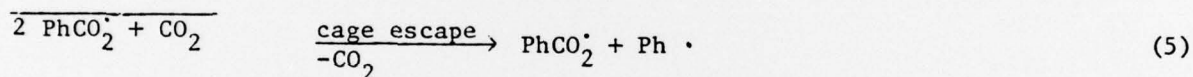
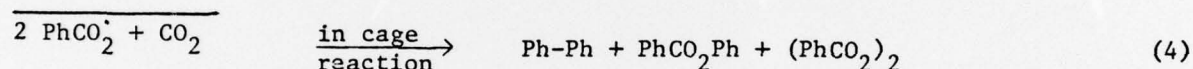
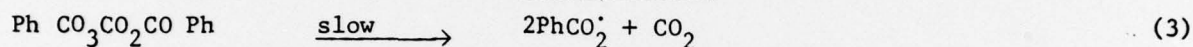
When the decomposition of Z is carried out in the presence of galvinoxyl at 79.3°C, the rate constant of reaction is $(1.22 \pm 0.01) \times 10^{-3} \text{ sec}^{-1}$. The absence of a significant inhibition of rate of the reaction of Z with galvinoxyl in the solution is inconsistent with a measurable induced decomposition path. Indeed, the slight acceleration of the reaction rate in the presence of galvinoxyl may signal the operation of an electron transfer induced reaction. We have observed similar electron transfer processes in the reaction of galvinoxyl and other peroxides and are continuing to examine this path.¹² Quantitative determination of the amount of galvinoxyl consumed during the thermolysis of Z was accomplished by spectrophotometry. These measurements show that at most 60% of the peroxy carbonate Z that reacts under these conditions gives cage escaped radicals. Consistent with this notion is the observation that the yield of biphenyl drops from 64% to 22% in the presence of galvinoxyl at 79.8°C. Thus, biphenyl must be a product of both in cage and cage escaped radicals. On the other hand, the yield of phenyl benzoate increases from ca. 6% to 20% when galvinoxyl is added to the reaction mixture. This change is consistent also with the partial intervention of an electron transfer path in the presence of galvinoxyl.

We investigated briefly the effect of solvent polarity on the thermolysis of Z . The rate constant for reaction of Z in CH_3CN at 79.8°C is $(9.64 \pm 1) \times 10^{-4} \text{ s}^{-1}$;

not significantly different from the benzene solvent value. Thus, we conclude that the carboxy inversion reaction, which is typically accelerated by polar solvents,¹³ does not contribute to the reaction of $\underline{2}$.

The thermolysis of peroxy carbonate $\underline{2}$ in benzene gives products quite similar to those obtained from the thermolysis of benzoyl peroxide. The formation of these products, and the observed kinetic behavior, can be understood in terms of the reactions in Scheme 1.

Scheme 1



Several points concerning this scheme warrant further discussion. The relatively low ΔS^\ddagger for reaction 3 is consistent with the direct expulsion of CO_2 forming simultaneously two benzoyloxy radicals.¹¹ Viewed in its simplest form, this reaction generates these radicals separated initially by a CO_2 molecule. The ratio of cage escape (eq. 4) to cage recombination (eq. 5) deduced from the galvinoxyl trapping experiment indicates that, despite the presence of the CO_2 , cage recombination in this case is significantly more efficient than for benzoyl peroxide (89% cage escape).¹⁴ This is contrary to the results of Kiefer and Traylor,¹⁸ and may indicate considerable motion within the cage before recombination or escape occurs.

In eq. 4 we indicate the possible formation of benzoyl peroxide as an in-cage product of the thermolysis of $\mathbf{2}$. Indeed, direct thermolysis of benzoyl peroxide does produce some in-cage regeneration of the reactant.¹⁵ However, we are not able to detect any benzoyl peroxide, either spectroscopically or kinetically, during the reaction of $\mathbf{2}$. Computer simulation of the reaction kinetics indicates that we would be unable to detect less than 5% of this product.

In summary, the ozonation of diphenylvinylene carbonate gives dibenzoylmonoperoxy carbonate, the first reported compound of its class. Its formation is consistent with the Criegee mechanism for the ozonation of olefins. The thermolysis of dibenzoylmonoperoxy carbonate produces the same radicals as benzoyl peroxide, but at a significantly greater rate. This might make $\mathbf{2}$ useful as a source of free radicals.

Acknowledgement: This work was supported in part by the National Science Foundation, and in part by the Office of Naval Research. We also thank Dr. Joseph Zupancic of this department for his many useful suggestions.

Experimental

General: IR spectra were recorded on a Perkin-Elmer 237B or a Perkin-Elmer 137 instrument. The ^1H NMR was determined on a Varian HR 220 instrument using an internal deuterium lock. The ^{13}C NMR was measured on a Varian XL 100, also with an internal deuterium lock. The acquisition time was 1.35 seconds and 56,000 transients were averaged. Visible absorptions were recorded with a Perkin-Elmer 101 instrument. Temperatures were measured with a NBS thermometer. The ozonation was done with a Welsbach ozonizer. Microanalysis was performed by J. Nemeth and Associates, University of Illinois. Diphenylvinylene carbonate ($\mathbf{1}$) was prepared by the method of Lewis and Hirsh.¹⁶

Dibenzoylmonoperoxy carbonate ($\mathbf{2}$): Diphenylvinylene carbonate (1.00 g, 4.20 mmol) was dissolved in 50 mL of CH_2Cl_2 and cooled in an ice bath. Ozone (9 mmol, 3 mol% in O_2) was bubbled in through a glass frit. After ozonation the solvent

was removed in vacuo, and the resulting solid was recrystallized from hexane, giving 0.908 g (3.17 mmol) dibenzoylmonoperoxycarbonate (75.6%): mp 87°C; ^1H NMR (220 MHz, CDCl_3) δ 8.10(d), δ 8.03(d), δ 7.35-7.70 (m); ^{13}C NMR (25.2 MHz, CDCl_3) δ 162.1, δ 158.9, δ 146.9, δ 124.6-135.2 (8 lines): IR (CCl_4) 1853 cm^{-1} , 1787 cm^{-1} ; Anal. Calcd. for $\text{C}_{15}\text{H}_{10}\text{O}_6$: C, 62.94%; H, 3.52%; active O, 5.59%, Found: C, 63.02%, H, 3.62%; active O, 5.57%.

Thermolysis of 2 in benzene: The products and yields were determined as follows: A 0.1278 g (0.447 mmol) sample of 2 was dissolved in 20 ml of benzene and heated at reflux for 2 hr. A small flow of N_2 was maintained at all times and the escaping gasses were passed through two dry ice/alcohol traps to remove solvent vapors and then into an ascarite trap. The ascarite gained 0.0404 g, which corresponds to 0.918 mmol CO_2 (yield 2.05 mol/mol peroxide). After cooling the reaction mixture, 0.0800 g of phenanthrene were added to the reaction mixture as an internal standard and comparison by GLC (6' SE-30 4%, 200°C) to a standard solution gave: biphenyl- 0.0442 g (0.287 mmol, 0.642 mol/mol peroxide), and phenyl benzoate- 0.00455 g (0.023 mmol, 0.051 mol/mol peroxide). The solvent was then removed in vacuo and the white solid which resulted was dried for 2 hr in vacuo. The mass of the residue was 0.242 g. This was dissolved in 20 mL 2-propanol and titrated to a phenolphthalein endpoint with 6.18 ml NaOH (0.0503 N). This corresponds to 0.311 mmol of benzoic acid. An extraction with NaOH of another sample had shown the acidic product to be benzoic acid. The residue not accounted for by biphenyl, phenyl benzoate, benzoic acid, and phenanthrene corresponds to 0.24 mmol when taken as tetrahydroquarterphenyl.

The rate constants for the thermal decomposition of 2 in benzene were determined as follows: 0.1274 g of 2 were placed in a sealed tube with 20 mL of benzene, and the tube was placed in a thermostated water bath. For samples at 60.00, 70.10, and 91.10°C, the method of Banerjee and Budke¹⁷ was used. Samples were taken at appropriate time intervals over two half-lives, and a 1 mL aliquot was diluted to 25 mL with a 2:1 mixture of acetic acid and CHCl_3 , and then purged with N_2 for 1 min

Finally, 1 mL 50% KI was then added and the visible absorbance of the triiodide formed was measured at 470 nm. For the samples at 66.02 and 75.90°C, the relative peroxide concentrations were determined by ir. A plot of the absorbance of the peak at 1787 cm⁻¹ for known concentrations of the peroxide against concentration was shown to be linear. Semilog plots of the absorbances determined by the above methods were made, and least squares analyses gave the rate constants and deviations listed in Table 2.

~~Thermolysis of 2 in the presence of galvinoxyl:~~ Galvinoxyl was obtained from Aldrich Chemical Company, and was shown to be pure by iodometric titration.¹⁸ For the kinetic analysis 6.81 mg (0.0238 mmol) of 2 and 18.98 mg (0.045 mmol) of galvinoxyl were dissolved in 25 mL of benzene. The visible absorbance of the galvinoxyl at 733 nm in this mixture was measured continuously. Importantly, galvinoxyl was shown not to decompose spontaneously under these conditions.

To determine the fraction of cage escape 0.02014 g (0.0477 mmol) of galvinoxyl and 0.0594 g (0.0208 mmol) of 2 were diluted to 25 mL with benzene and purged with N₂ for 5 min. The absorbance of this solution was measured at 740 nm. Samples were then placed in three tubes, purged for an additional minute, and sealed. After reaction for 1.5 h at 97.90°C, the absorbances were again measured, and the amount of galvinoxyl consumed was determined.

Table 1: Products of Thermolysis of 2 in Benzene at 81.1°C.

Product	Yield ^a	CO ₂ balance ^b	Radical balance ^c
CO ₂	2.0	2.0	-----
Biphenyl	0.64	-----	1.28
Phenyl benzoate	0.05	0.05	0.10
Benzoic acid	0.69	0.69	-----
Residue ^d	0.54	-----	1.08
Totals		2.74	2.46

^a in mol product/ mol peroxide.

^b in mol CO₂ or Carboxyl groups/mol peroxide

^c in mol radicals incorporated/mol peroxide

^d as tetrahydroquarterphenyl

Table 2: Rate constants for thermolysis in benzene.

Temp. (°C)	k ₁ x 10 ⁵ (sec ⁻¹)
60.00	8.05 ± 0.55
66.02	19.2 ± 0.6
70.10	26.3 ± 2.5
74.90	49.5 ± 2.5
81.10	111 ± 2

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